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2.	Patent application number *(The Patent Office will fill in this part)*	**9918117.4**
3.	Full name, address and postcode of the or of each applicant *(underline all surnames)*	ACMA LIMITED (formerly Tioxide Specialties Limited) The Heath Runcorn Cheshire WA7 4QF
	Patents ADP number *(if you know it)*	6074082001 7667371001MB.
	If the applicant is a corporate body, give the country/state of its incorporation	UNITED KINGDOM
4.	Title of the invention	ORGANOMETALLIC COMPOSITIONS AND POLYISOCYANATE COMPOSITIONS CONTAINING THEM
5.	Name of your agent *(if you have one)*	~~JACKSON, JOHN DEREK~~
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11. I/We request the grant of a patent on the basis of this application.

Signature John Derek Jackson

Date 2nd August 1999

12. Name and daytime telephone number of person to contact in the United Kingdom

JOHN DEREK JACKSON
01642 437419

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ORGANOMETALLIC COMPOSITIONS AND POLYISOCYANATE COMPOSITIONS CONTAINING THEM

This invention relates to novel organometallic compositions and polyisocyanate compositions containing them and, in particular, to organometallic compositions based on iron or aluminium and which are useful in polyisocyanate compositions for binding lignocellulosic material.

The use of organic polyisocyanates as binders for lignocellulosic material in the manufacture of sheets or moulded bodies such as waferboard, chipboard, fibreboard and plywood is well known. In a typical process the organic polyisocyanate, optionally in the form of a solution, dispersion or aqueous emulsion, is applied to the lignocellulosic material which is then subjected to heat and pressure.

Suitable polyisocyanate compositions are disclosed in PCT Application WO 97/17388 and our co-pending UK Application GB 9815029.5. These compositions comprise a Group IVB metal compound, preferably a titanium chelate, optionally in combination with a compatibilising compound and/or conventional release agents. Although these compositions perform well as binders for lignocellulosic material and provide good release performance, it is desirable to develop alternative compositions which provide at least as good stability on storage before use, together with good curing properties and excellent bonding strength when applied to the lignocellulosic material.

It has now been surprisingly found that certain compositions prepared from β–dicarbonyl compounds can be used to prepare polyisocyanate compositions which are very stable on prolonged storage and economical when used for binding lignocellulosic material.

According to one aspect of the invention an organometallic composition, suitable for use in curing polyisocyanate compositions, comprises a complex of

at least one metal selected from iron and aluminium and at least one β-dicarbonyl compound wherein the molar ratio of metal to β-dicarbonyl compound is in the range 1 : 3.5 to 1 : 10.

Also according to the invention, a polyisocyanate composition comprises an organometallic composition, said organometallic composition being a complex of at least one metal selected from the group consisting of iron and aluminium and a β-dicarbonyl compound in which the molar ratio of metal to β-dicarbonyl compound is in the range 1 : 3.5 to 1:10 .

The iron or aluminium composition of the invention, suitable for use in a polyisocyanate composition, is described herein as a "complex". It is believed that some of the β-dicarbonyl compound will be chemically bound to the metal (Fe or Al) but some can be described as "free" ester. The exact proportions which are bound and free will depend partly upon the exact molar ratios present in the complex and which metal is used, but it has been shown that the "free" ester does influence the properties, particularly the stability on storage, of a polyisocyanate composition as a binder for lignocellulosic materials. The use of the word "complex" does not imply that said complex is necessarily separately prepared before addition to a polyisocyanate to form the polyisocyanate compositions of the invention. The complex can be formed in the course of preparing the inventive polyisocyanate composition using alternative methods as described hereinafter.

The β-dicarbonyl compound used to prepare the complexes of the invention can be any suitable β-dicarbonyl compound. Preferred β-dicarbonyl compounds include acetylacetone (2,4-pentanedione), benzoyl acetone, dibenzoylmethane, 2,2,6,6-tetramethylheptanedione and 1,1,1-trifluoro-2,4-pentanedione and esters of acetoacetic acid such as ethylacetoacetate,

methylacetoacetate, isopropylacetoacetate and tertiarybutylacetoacetate. A complex may be based on one β-dicarbonyl compound but particularly useful complexes have been prepared from a mixture of β-dicarbonyl compounds, such as a mixture of acetylacetone and ethylacetoacetate.

The molar ratio of metal to β-dicarbonyl compound in the complex is in the range 1 : 3.5 to 1 : 10. Preferably, the molar ratio is preferably in the range 1 : 4 to 1 : 8 and more preferably in the range 1 : 5 to 1 : 8. In agreement with conventional theories about the co-ordination chemistry of iron or aluminium, it is believed that three molecules of the β-dicarbonyl compound will be chemically bound to a metal atom and the remainder will be "free".

Typically, the complexes of iron or aluminium are prepared from aluminium or iron alkoxides having the general formula $M(OR)_3$ in which M is Al or Fe and R is a substituted or unsubstituted, cyclic or linear, alkyl, alkenyl, aryl or alkyl-aryl group or mixtures thereof. Preferably, R contains up to 8 carbon atoms and, more preferably, up to 6 carbon atoms. Generally, all three OR groups will be identical but alkoxides derived from a mixture of alcohols can be used and mixtures of alkoxides can be employed when more than one metal is present in the complex. Suitable alkoxides include trimethoxyaluminium, trimethoxyiron, triethoxyaluminium, triethoxyiron, tri-isopropoxyaluminium, tri-isopropoxyiron, tri-n-propoxyaluminium, tri-n-propoxyiron, tritertiarybutoxy-aluminium, tritertiarybutoxyiron, tri-sec-butoxyaluminium and tri-sec-butoxyiron.

Alternatively, the complex can be prepared from compounds known as condensed alkoxides of aluminium. A condensed alkoxide is typically prepared by the controlled addition of water to an alkoxide, followed by removal of alcohol which is displaced. The condensed alkoxides which are produced consist of a mixture of polymeric compounds containing Al-O-Al linkages and AlOR groups in which R has the meaning given above. Complexes of condensed aluminium

alkoxides can also be prepared by forming a complex of a β-dicarbonyl compound with an alkoxide, adding water to the complex and removing any by-product alcohol.

Other aluminium or iron compounds, such as aluminium or iron trichloride or alkoxides which have been substituted with other moieties can be used as raw materials for the formation of the complex used in the invention.

The complex can be readily prepared by mixing, for example, an alkoxide or condensed alkoxide with an appropriate amount of β-dicarbonyl compound. Alcohol from the alkoxide will be displaced by the β-dicarbonyl compound and, preferably, the displaced alcohol is removed by, for example, distillation. In a preferred method, 3 moles of β-dicarbonyl compound (e.g. acetylacetone) per atom of Al or Fe are added to an alkoxide or condensed alkoxide and the displaced alcohol is removed by distillation. Any additional β-dicarbonyl compound required is then added to the stripped product. This method is advantageous because it provides a consistent product of known stoichiometry.

In one method of preparing the polyisocyanate compositions of the invention, a product containing 2 or 3 moles of β-dicarbonyl compound per Fe or Al atom can be mixed with additional β-dicarbonyl compound to produce the complex of the invention and this complex can be added to a polyisocyanate composition. For aluminium complexes, a product containing 1 mole of β-dicarbonyl compound per Al atom is a suitable starting material for this method. Alternatively, the product containing 2 or 3 moles of β-dicarbonyl compound per Fe or Al atom can be prepared and any additional β-dicarbonyl compound required to produce the polyisocyanate composition of the invention can be added to the polyisocyanate before or after the iron or aluminium

compound has been added. Other methods of preparing the composition of the invention will be apparent to a person skilled in this art.

The amount of iron or aluminium complex present in the polyisocyanate composition of the invention is usually in the range 0.01 to 20 % by weight, based on the polyisocyanate and, preferably, the amount is in the range 0.1 to 10%. More preferably, the amount of complex present is in the range 0.2 to 7% by weight with respect to polyisocyanate and, frequently, acceptable curing can be achieved if the amount of complex present is in the range 0.2 to 2% by weight with respect to polyisocyanate.

Polyisocyanates for use in the polyisocyanate composition of the present invention may be any organic polyisocyanate compound or mixture of organic polyisocyanate compounds, provided said compounds have at least 2 isocyanate groups. Organic polyisocyanates include diisocyanates, particularly aromatic diisocyanates, and isocyanates of higher functionality.

Examples of organic polyisocyanates which may be used in the composition of the present invention include aliphatic isocyanates such as hexamethylene diisocyanate; and aromatic isocyanates such as m- and p-phenylene diisocyanate, tolylene-2,4- and tolylene-2,6-diisocyanate, diphenylmethane-4,4'-diisocyanate, chlorophenylene-2,4-diisocyanate, naphthylene-1,5-diisocyanate, diphenylene-4,4'-diisocyanate, 4,4'-diisocyanate 3,3'-dimethyldiphenyl, 3-methyldiphenylmethane-4,4'-diisocyanate and diphenyl ether diisocyanate; and cycloaliphatic diisocyanates such as cyclohexane-2,4- and cyclohexane-2,3-diisocyanate, 1-methylcyclohexyl-2,4- and 1-methylcyclohexyl-2,6-diisocyanate and mixtures thereof and bis-(isocyanatocyclohexyl)methane and triisocyanates such as 2,4,6-triisocyanatotoluene and 2,4,4-triisocyanatodiphenylether.

Modified polyisocyanates containing isocyanurate, carbodiimide or uretonimine groups may be employed as well. Further, blocked polyisocyanates, like the reaction product of a phenol or an oxime and a polyisocyanate, may be used, having a deblocking temperature below the temperature applied when using the polyisocyanate composition.

The organic polyisocyanate may also be an isocyanate-ended prepolymer made by reacting an excess of a diisocyanate or higher functionality polyisocyanate with a polyol.

Water-emulsifiable organic polyisocyanates like those described in UK patent no. 1 444 933, in European patent publication no. 516 361 and in PCT patent publication no. 91/03082 can also be used.

Mixtures of isocyanates may be used, for example a mixture of tolylene diisocyanate isomers such as the commercially available mixtures of 2,4- and 2,6-isomers and also the mixture of di- and higher poly-isocyanates. Polyisocyanate mixtures may optionally contain monofunctional isocyanates such as p-ethyl phenylisocyanate.

Such mixtures are well-known in the art and include the crude phosgenation products containing methylene bridged polyphenyl polyisocyanates, including diisocyanate, triisocyanate and higher polyisocyanates together with any phosgenation by-products.

Preferred isocyanates to be used in the present invention are those wherein the isocyanate is an aromatic diisocyanate or polyisocyanate of higher functionality such as a pure diphenylmethane diisocyanate or a mixture of methylene bridged polyphenyl polyisocyanates containing diisocyanates, triisocyanates and higher functionality polyisocyanates.

Methylene bridged polyphenyl polyisocyanates are well known in the art. They are prepared by phosgenation of corresponding mixtures of polyamines.

For convenience, polymeric mixtures of methylene bridged polyphenyl polyisocyanates containing diisocyanate, triisocyanate and higher functionality polyisocyanates are referred to hereinafter as polymeric MDI. Suitable polyisocyanates include SUPRASEC™ DNR, SUPRASEC™ 2185, RUBINATE™ M and RUBINATE™ 1840, all available from Huntsman Polyurethanes.

Preferably the polyisocyanate is liquid at room temperature.

Conventional release agents can be added to or used in combination with the polyisocyanate composition of the present invention containing an iron or aluminium complex of a β-dicarbonyl compound.

The conventional release agent is present in an amount varying between 0.2 and 10 %, preferably between 0.5 and 6 % and most preferably between 1 and 3 % by weight based on the polyisocyanate whereas the iron or aluminium complex of a β-dicarbonyl compound is preferably present in an amount varying between 0.2 and 4 %, most preferably between 0.2 and 2 % by weight based on the polyisocyanate.

Examples of conventional release agents include polysiloxanes, saturated or unsaturated fatty acids (such as oleic acid) or fatty acid amides or fatty acid esters and polyolefin waxes.

Preferred conventional release agents to be used according to the present invention are polyolefin waxes or mixtures of polyolefin waxes, especially functionalised polyolefin waxes, which are dispersible in an aqueous medium to form an aqueous emulsion. More preferably, the polyolefin waxes are selected from oxidised polyethylene waxes and oxidised polypropylene waxes.

A preferred method for using the release agent is to apply the emulsion to the surface of the polyisocyanate treated lignocellulosic material or to the press metal surface prior to hot pressing the combination.

When used, the aqueous emulsion of the polyolefin wax should normally contain a sufficient amount of the polyolefin wax to provide a coverage of about 0.01 to about 1, and preferably about 0.02 to about 0.5 mg of the polyolefin wax per cm^2 of lignocellulosic material or press metal surface. Generally, lower levels of polyolefin wax are preferred as they are more cost effective. When taking the emulsifiers into account, the aqueous emulsions will usually contain about 0.2 to about 10 %, preferably about 0.3 to about 5 % by weight of total solids. The emulsions are usually prepared at 30 to 40 % total solids, transported to the point of use and then diluted with water to the desired concentration.

It has been found that the polyolefin wax emulsion, when used in combination with polyisocyanate compositions of the present invention, may be usefully applied to the lignocellulosic material or press metal surface in an amount equivalent to 8 to 14 mg per cm^2.

A particularly preferred polyethylene wax emulsion which can be used in a process in combination with a polyisocyanate composition of the present invention is Rubilon™ 603 or Rubilon™ 605 , both available from Huntsman Polyurethanes.

A particularly preferred polypropylene wax emulsion which can be used in the present process is ME 42040 available from Michelman Inc. of Cincinnati, Ohio.

In order to further improve the storage stability of the polyisocyanate composition of the present invention a diluent may be added to the composition. Suitable diluents include plasticizers of the type mentioned in "Taschenbuch der

Kunststoff-Additive", Ed. by R. Gachter and H. Muller, Carl Hanser Verlag München, third edition, 1989. Preferred diluents are phthalates, aliphatic carboxylates, fatty acid esters, linseed oil and soybean oil. A particularly preferred diluent is Priolube 1403 available from Unichema being methyl oleate. These diluents are added in amounts of from 1 to 40 parts by weight per 100 parts by weight of polyisocyanate and preferably in amounts of from 1 to 15 parts by weight per 100 parts by weight of polyisocyanate.

The composition further may comprise conventional additives like flame retardants, lignocellulosic preserving agents, fungicides, waxes, sizing agents, fillers, surfactants, thixotropic agents and other binders like formaldehyde condensate adhesive resins and lignin (optionally in combination with a lignin solvent such as described in PCT Patent Application No. EP96/00924).

A particularly preferred additive to be used in the polyisocyanate composition of the present invention is a coupling agent such as an organofunctional silane (for example, Dynasylan AMEO, available from Huels). Adding such a coupling agent to the polyisocyanate composition leads to improved board properties. The organofunctional silane coupling agents are used in amounts ranging from 0.01 to 3 %, preferably from 0.1 to 2 % by weight based on the polyisocyanate.

The polyisocyanate composition of the present invention can be made by simply mixing the ingredients at room temperature.

The present invention is also concerned with a process for preparing lignocellulosic bodies by bringing lignocellulosic parts into contact with the present polyisocyanate composition and by pressing this combination.

Therefore the present invention also provides a process for binding lignocellulosic material comprising the steps of

a) bringing said lignocellulosic material in contact with the polyisocyanate composition according to one embodiment of this invention and

b) subsequently allowing said material to bind.

The lignocellulosic bodies are prepared by bringing the lignocellulosic parts into contact with the polyisocyanate composition by means such as mixing, spraying and/or spreading the composition with/onto the lignocellulosic parts and by pressing the combination of the polyisocyanate composition and the lignocellulosic parts, preferably by hot-pressing, normally at 150° C to 250°C and 2 to 6 MPa specific pressure.

Such binding processes are commonly known in the art.

In waferboard manufacture the lignocellulosic material and the polyisocyanate composition may be conveniently mixed by spraying the present polyisocyanate composition on the lignocellulosic material while it is being agitated.

As described hereinbefore, in a preferred process according to the invention, a release agent, which is preferably an aqueous emulsion of a polyolefin wax, is applied to the surface of the polyisocyanate treated lignocellulosic material or to the press metal surface prior to hot pressing the combination.

The lignocellulosic material after treatment with the polyisocyanate composition is placed on caul plates made of aluminium or steel which serve to carry the furnish into the press where it is compressed to the desired extent usually at a temperature between 150° C and 250° C.

While the process is particularly suitable for the manufacture of waferboard known extensively as oriented strand board and will be largely used for such manufacture, the process may not be regarded as limited in this

respect and can also be used in the manufacture of medium density fiberboard, particle board (also known as chipboard) and plywood.

Thus the lignocellulosic material used can include wood strands, woodchips, wood fibres, shavings, veneers, wood wool, cork, bark, sawdust and like waste products of the wood working industry as well as other materials having a lignocellulosic basis such as paper, bagasse, straw, flax, sisal, hemp, rushes, reeds, rice hulls, husks, grass, nutshells and the like. Additionally, there may be mixed with the lignocellulosic materials other particulate or fibrous materials such as ground foam waste (for example, ground polyurethane foam waste), mineral fillers, glass fibre, mica, rubber, textile waste such as plastic fibres and fabrics.

When the polyisocyanate composition is applied to the lignocellulosic material, the weight ratio of polyisocyanate/lignocellulosic material will vary depending on the bulk density of the lignocellulosic material employed. Therefore, the polyisocyanate compositions may be applied in such amounts to give a weight ratio of polyisocyanate/lignocellulosic material in the range of 0.1:99.9 to 20 : 80 and preferably in the range of 0.5 : 99.5 to 10 : 90.

If desired, other conventional binding agents, such as formaldehyde condensate adhesive resins, may be used in conjunction with the polyisocyanate composition.

More detailed descriptions of methods of manufacturing waferboard and similar products based on lignocellulosic material are available in the prior art. The techniques and equipment conventionally used can be adapted for use with the polyisocyanate compositions of the present invention.

The polyisocyanate compositions of the present invention are extremely effective in minimising unwanted adhesion to caul plates, press plates and other surfaces with which the treated lignocellulosic material may come into contact.

Their storage stability and release performance is improved compared to polyisocyanate compositions of the prior art, as well as the obtained board properties.

The sheets and moulded bodies produced from the polyisocyanate compositions of the present invention have excellent mechanical properties and they may be used in any of the situations where such articles are customarily used.

The invention is illustrated but not limited by the following examples.

EXAMPLE 1

Aluminium tris(acetylacetonate)

To a flask containing 50 g of a mixture of heptane isomers was added 30.8 g (0.125 mol) of Al tris(sec-butoxide). To this was added with stirring 37.55g (0.375 mol) of acetylacetone. After stirring, a white precipitate of aluminium tris(acetylacetonate) was collected and washed on a filter paper. Yield = 36.6 g (91 %).

Iron tris(acetylacetonate)

20.3 g of anhydrous iron(III) chloride (0.124 mol) was dissolved in 30 g of demineralised water with gentle warming. To this was added 45 ml of ammonia solution (SG. 0.880). The mixture was then heated on a steam bath for 30 minutes. The precipitate of iron(III) hydroxide was filtered and washed free of chloride. The moist iron (III) hydroxide and 60 g of acetylacetone (0.6 mol) were mixed together and heated for 30 minutes. Crystals of $Fe(acac)_3$ were deposited and were recrystallised from ethanol.

The metal acetylacetonates prepared as above were mixed with ethylacetoacetate (EAA) in various molar ratios, as shown in Table 1.

TABLE 1

Sample	Metal	Moles EAA added*
1A	Fe	0.5
1B	Fe	2.0
1C	Fe	4.0
1D	Al	0.5
1E	Al	4.0

* per mole metal tris(acetylacetonate)

The samples were evaluated by preparing a number of compositions comprising 100 parts by weight of polyisocyanate (polymeric MDI, Suprasec DNR, available from Huntsman Polyurethanes) and 0.3 parts of the samples designated 1A to 1F or (for comparison) iron or aluminium tris(acetylacetonate). The compositions were then stored at 45° C and the viscosity tested by means of a Brookfield viscometer at various intervals. Results are given in Table 2 below [all viscosities are in Pa s].

TABLE 2

Time (days)	Sample						
	1A	1B	1C	1D	1E	$Fe(AcAc)_3$	$Al(AcAc)_3$
0	202	202	202	202	202	202	202
10	250	250	262	224	220	474	344
21	376	300	334	251	242	1030	425
70	978	542	509	536	258	3602	615
94	1736	704	732	406	308	6448	880

Generally, the most relevant period for storage stability at room temperature is the period 14 to 46 days after production of the polyisocyanate composition. From Table 2 it can be seen that iron and aluminium complexes of the invention used in polyisocyanate compositions of provide an economical means of stabilising the compositions.

EXAMPLE 2

To a flask containing 50.02 g of a mixture of heptane isomers was added 31.0 g (0.125 mol) of aluminium tris(sec-butoxide). To this was added, with stirring, 48.87 g (0.375 mol) of ethylacetoacetate. After stirring the liquor was transferred to a rotary evaporator flask and the volatile sec-butanol and heptanes (78.66 g) were removed under vacuum leaving aluminium tris(ethylacetoacetate) (51.23 g, 0.125 mol).

Samples of aluminium tris(ethylacetoacetate) prepared as above was mixed with additional ethylacetoacetate as indicated in Table 3 below.

TABLE 3

Sample	Moles EAA added*
2A	0.5
2B	4.0

*per mole of $Al(EAA)_3$

The samples were evaluated by preparing compositions comprising 100 parts by weight of polyisocyanate (polymeric MDI, Suprasec DNR, available from Huntsman Polyurethanes) and 0.3 parts of the samples designated 2A and 2B or (for comparison) aluminium tris(ethylacetoacetate). The compositions were then stored at 45° C and the viscosity tested by means of a Brookfield viscometer at various intervals. Results are given in Table 4 below [all viscosities are in Pa s].

TABLE 4

Time (days)	Sample		
	2A	2B	$Al(EAA)_3$
0	224	224	224
10	236	234	272
20	258	252	310
49	256	274	324
73	290	418	510

EXAMPLE 3

A sample of an organometallic composition was prepared by mixing 1 mole of iron tris(acetylacetonate), as prepared in Example 1, with a further 2.5 moles ethylacetoacetate. The sample was evaluated by preparing a composition comprising 100 parts by weight of polyisocyanate (polymeric MDI, Rubinate 1840, available from Huntsman Polyurethanes) and 0.3 parts of the sample. The composition was then stored at 45° C and the viscosity tested by means of a Brookfield viscometer at various intervals. Results are given in Table 5 below [all viscosities are in Pa s].

TABLE 5

Time (days)				
0	20	40	65	100
220	290	300	420	620

EXAMPLE 4

An organometallic complex having a composition similar to that in Example 3 was prepared by mixing 2.5 g of iron tris(acetylacetonate), available from Aldrich, with 2.3 g ethylacetoacetate in a flask with stirring.

The organometallic complex (0.48 parts) was mixed with 100 parts by weight of polyisocyanate (polymeric MDI, SUPRASEC DNR, available from Huntsman Polyurethanes) .

This composition was used to prepare single layer boards of aspen at 2.5% and 6% resin based on dry wood. Boards of 12 x 450 x 450 mm and density 650 kg/m^3 were made using the above composition and also containing a sizing wax emulsion (SPG 60 available from Condea Chemie) at 1 % loading (solid wax) based on dry wood. The prepressing moisture content was 8 %. For comparison, similar boards were prepared using the polyisocyanate without the organometallic complex (Comparison).

Composites where then prepared using the following pressing conditions:

Press Close (Contact position):	50s
Cure Time:	10s / mm
Degas Time:	15s
Press Temperature:	200° C

The composites produced under conditions described above were then tested using a range of industry standard tests to measure physical properties (Modulus of elasticity and rupture according to EN310 ; Swelling after 24hrs of soaking in water according to EN317). Results are given in Table 6 below.

TABLE 6

	2.5% Binder loading				6% Binder loading			
Property	Example 4	SD	Comparison	SD	Example 3	SD	Comparison	SD
Modulus of Elasticity (%)	4562	797	4993	431	5345	1129	5113	644
Modulus of Rupture (%)	34.9	9.0	35.5	5.8	44.5	12.7	43.5	9.0
Thickness Swell (%)	16.5	1.6	17.0	3.3	10.6	1.0	7.8	1.0

The measured properties showed no significant performance difference between composites prepared with SUPRASEC DNR alone and when the product of Example 4 was present. This demonstrates the utility of using polyisocyanate compositions containing compositions of the invention to produce composites without affecting physical performance of the resulting composite panels.

EXAMPLE 5

Boards were made using a polyisocyanate comprising different parts by weight of the organometallic complex of Example 4 and 100 parts by weight of standard polyisocyanate (SUPRASEC DNR – see Table 7). The release performance of the boards was tested. In order to reproduce the failure mode all spray of release agent was stopped from the 7th repeat onwards. It is considered that a release rating (see below) of 3 will cause a catastrophic failure under plant conditions. The release trials were run in parallel and for each composite the release from the top and bottom platten was measured. The per cent wood failure (amount of the platten covered by residual wood) was also recorded. Results are summarised in Tables 8 and 9 below.

Composites where prepared using the following pressing conditions:

Press Close (Contact position):	20s
Cure Time:	9.3s / mm
Degas Time:	20s
Press Temperature:	170° C

TABLE 7

Sample	Weight per cent Example 4 product	Release agent (g/m²)
5A	0	4
5B	0.48	2
5C	0.32	2
5D	0.32	1

TABLE 8

No. of repeats	Release Rating		%Wood Failure		Release Rating		% Wood Failure	
	5A Top	5A Bottom	5A Top	5A Bottom	5B Top	5B Bottom	5B Top	5B Bottom
1	4.5	5	0.5	5	5	5	0	0
2	5	4.5	0.5	7	5	5	0	0
3	5	4	2	10	5	5	0	0
4	5	4.5	3	10	5	5	0	0
5	5	5	3	10	5	5	0	0
6	5	4	1	12	5	5	0	0
7	5	4	2	15	5	5	0	0
8	4.5	5			5	5		
9	4.5	5			5	5		
10	4.5	5	2	15	4.5	5	0	0.5

TABLE 9

No. of repeats	Release Rating		%Wood Failure		Release Rating		% Wood Failure	
	5C Top	5C Bottom	5C Top	5C Bottom	5D Top	5D Bottom	5D Top	5D Bottom
1	5	5	0	0	5	5	0	0
2	5	5	0	0	5	5	0	0
3	5	5	0	0	5	5	0	0.5
4	5	5	0	0	5	5	0	0
5	5	5	0	0	5	5	0	0
6	5	4.5	0	0	4.5	5	0	0
7	5	4.5	0	0	5	5	0	0
8	5	5			4.5	5		
9	4.5	5			5	5		
10	4	5	0.5	0	4.5	5	0	0

The release rating is given a value from 1 to 5 having the following significance:

1	complete sticking, board can not be removed without destruction of the board
2	sticking with wood failure higher than 50 %
3	sticking with wood failure less than 25 % but higher than 5 %
4	sticking with wood failure less than 5 %. Little force needed to remove board.
4.5	sticking without wood failure, hanging board. No effort needed to remove board.
5	perfect release, the board releases spontaneously.

The results of Table 7 and 8 show that a composition containing an organometallic composition of the invention improves the performance of a system using an external release agent and allows this performance to be maintained as the level of external release agent is significantly reduced.

CLAIMS

1. An organometallic composition, suitable for use in curing polyisocyanate compositions, comprising a complex of at least one metal selected from iron and aluminium and at least one β-dicarbonyl compound wherein the molar ratio of metal to β-dicarbonyl compound is in the range 1 : 3.5 to 1 : 10.

2. An organometallic composition according to claim 1 characterised in that the molar ratio of metal to β-dicarbonyl compound is in the range 1 : 4 to 1 : 8.

3. An organometallic composition according to claim 1 or 2 characterised in that the β-dicarbonyl compound is acetylacetone, benzoyl acetone, dibenzoylmethane, 2,2,6,6-tetramethylheptanedione, 1,1,1-trifluoro-2,4-pentanedione, ethylacetoacetate, methylacetoacetate, isopropylaceto-acetate or tertiarybutylacetoacetate.

4. An organometallic composition according to any one of the preceding claims characterised in that the complex is derived from a condensed alkoxide of aluminium.

5. A polyisocyanate composition comprising an organometallic composition according to any one of the preceding claims.

6. A polyisocyanate composition according to claim 5 characterised in that the amount of organometallic composition present is in the range 0.01 to 20 per cent by weight.

7. A polyisocyanate composition according to claim 5 or 6 characterised in that the polyisocyanate is diphenylmethane diisocyanate or a mixture of methylene bridged polyphenyl polyisocyanates.

8. A polyisocyanate composition according to any one of claims 5 to 7 characterised in that the composition contains a release agent in an amount in the range 0.2 to 10 per cent by weight.

9. A polyisocyanate composition according to claim 8 characterised in that the release agent is a polysiloxane, a fatty acid, a fatty acid amide, a fatty acid ester or a polyolefine wax.

10. A polyisocyanate composition according to any one of claims 5 to 9 characterised in that the composition contains a diluent.

11. A process for binding lignocellulosic material comprising the steps of

a) bringing said lignocellulosic material in contact with a polyisocyanate composition according to any one of claims 5 to 10, and

b) subsequently allowing said material to bind.

12. A process according to claim 11 characterised in that the polyisocyanate composition is brought into contact with the lignocellulosic material and the combination thereby formed is hot-pressed at a temperature in the range 150°C to 250° C and 2 to 6 MPa specific pressure.

13. A process according to claim 11 or 12 characterised in that the polyisocyanate composition is applied in an amount to give a weight ratio of polyisocyanate to lignocellulosic material in the range 0.1 : 99.9 to 20 : 80.

14. A process according to any one of claims 11 to 13 characterised in that a release agent is applied to the surface of the polyisocyanate treated lignocellulosic material or to the press metal surface prior to hot pressing the combination.

ABSTRACT

An organometallic composition, suitable for use in curing polyisocyanate compositions, comprises a complex of at least one metal selected from iron and aluminium and at least one β-dicarbonyl compound wherein the molar ratio of metal to β-dicarbonyl compound is in the range 1 : 3.5 to 1 : 10. Also, a polyisocyanate composition comprises an organometallic composition, said organometallic composition being a complex of at least one metal selected from the group consisting of iron and aluminium and a β-dicarbonyl compound in which the molar ratio of metal to β-dicarbonyl compound is in the range 1 : 3.5 to 1:10 . A process for binding lignocellulosic material comprises the steps of a) bringing said lignocellulosic material in contact with the polyisocyanate composition according to one embodiment of this invention and, b) subsequently allowing said material to bind.